Chiang Mai J. Sci. 2009; 36(3) : 349-358
www.science.cmu.ac.th/journal-science/josci.html
Contributed Paper

Optimizing the Injection Parameter of Water Atomised SS316L Powder with Design of Experiment Method for Best Sintered Density

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Received: 29 March 2009
Accepted: 31 May 2009

Abstract

Injection moulding parameters that simultaneously satisfy green part qualities (green defect, green strength and green density) have been optimised using L_{27} (3^{13}) Taguchi orthogonal array. Moulding variables involved in the optimisation are the moulding pressure, moulding temperature, mould temperature, holding pressure, moulding rate, holding time and cooling time. The analysis of variance (ANOVA) is employed to determine the significant levels (α) and contributions of the variables to the green qualities. Results show that the mould temperature and holding time is highly significant simultaneously to the green qualities, while the holding pressure and cooling time does not show any significance. Besides that, densification of the compact moulded with the optimised moulding parameter is presented in this paper. With sintering temperature ranging from 1,340 to 1,400^{o}C while heating rate and dwell time remains at 10^{o}C/min and 4 hours respectively demonstrates that the optimised moulding parameter is enabled to produce a sintered part with a density which is very close to the solid density of SS316L.

Keywords: SS316L, taguchi method, analysis of variance, simultaneous optimisation, sintering.

1. Introduction

Metal injection moulding (MIM) has acquired an increasing importance as a production technique for small, complex stainless steel components [1, 2]. The technique involves mixing of the metal powder with a binder, injection of the mixture into the mould, removal of the binder and then sintering the end product to consolidate it to its final density [3]. Optimisation of each of these process procedures and appropriate selection of the starting materials, i.e. the powder and binder are considered critically important to the overall success of the process.

The determination and optimisation of the process parameters have triggered numerous research works, as they need to grasp the in depth knowledge of different processes and accurate modelling techniques for each stage. The traditional approach was where the experimental work had to verify one factor at a time, holding all other factors as fixed. This method did not produce satisfactory results even in a wide range of experimental settings. Barriere et al. [4], Omar [5], Ismail [6], Murtadhahadi [7] and Chuankrerkkul et al. [8] have been using classical Design of Experiment (DOE) technique to study the effects of moulding parameters on the green part quality such as green density, green strength and green defects. However, author of literature [9] studied the influence of injection moulding parameters on the properties of green parts using DOE technique. Taguchi method has been chosen to study the influence of injection parameter to the weight and dimension of the green part. Variables studied consist of injection speed, mould temperature, material temperature, holding pressure, holding pressure duration and cycle time. Two factor levels were used for each variable. The analysis of variance (ANOVA) presented by Berginc et al. [9] discovered that the injection parameter has a big influence to the weight and dimension of the green part and the mould temperature, material temperature and holding pressure are the variables which influence the dimensions most.

On the other hand, this paper present authors investigation on the influence of a different variables than that presented by Berginc et al. [9], such as moulding pressure, moulding temperature, mould temperature, holding pressure, moulding rate, holding time and cooling time. Three factor levels are used in this present paper instead of two factor level used by Berginc et al. [9]. This is to ensure the accuracy of the experimental result. In addition, green parts properties such as defects, strength and density are the quality characteristic are to be optimised. Finally, simultaneous injection parameter that able to produce stronger green parts and higher green density with fewer defects will be the injection parameter for producing green parts for the sintering parameter investigation. The aim of the injection parameter optimisation is to improve the sintered part density by improving the injection parameter by optimisation via DOE approach. This is because, from other experiments and in other areas of study such as plastic moulding, metal removal processes, the Taguchi method is recognised as a systematic application of design and analysis of experiments for the purpose of designing and improving product quality [10]. In recent years, the Taguchi method has become a powerful tool for improving productivity during research and development [11] so that high quality products can be produced in a short period of time and at low cost.

The objective of this paper is to optimise the moulding parameters that simultaneously satisfy a green part quality required for the MIM compact before it undergoes a sintering process to attain its mechanical properties. Less defects, high green strength and green density have been identified as the green part quality or as an output for this study [5]. An analysis of variance (ANOVA) is utilized to identify the significant level of each variable. The paper examines and discusses interactions of major moulding variables, such as moulding temperature, moulding pressure and mould temperature which influences the green part quality. Simultaneous optimisation for the entire quality characteristics is presented in the later part of the paper. The simultaneous optimisation is vital, as manufacturers prefer only a single moulding parameter that best fits both quality characteristics instead of using different sets of moulding parameters. Furthermore, as sintering is critical for determining the final quality of the parts produced, this paper presents a densification of the compacts moulded using the optimised parameter obtained in the optimisation process.

2. METHODOLOGY

A MPIF 50 standard tensile bar is used as a specimen. A 316L stainless steel water-atomised powder with pycnometer density of 7.90 g/cm^3 is mixed with 73% PEG weight of polyethylene glycol (PEG) and 25% weight of Polymethyl Methacrylate (PMMA). About 2% weight of stearic acid (SA) is used as a surfactant. The morphology and particle size distribution of the water atomised powder is shown in Figure 1 and Table 1 respectively. The powder loading for the coarse and fine powder feedstock is 63.5% volume and 62.5% respectively.

Prior to the moulding, compositions are mixed in a sigma blade mixer for 95 minutes at a temperature of 70^{o}C. Battenfeld, BA 250 CDC injection moulding machine is used to prepare the greens while the high vacuum furnace Korea VAC-TEC, VTC 500HTSF with vacuum pressure up to 9.5×10^{-6} mbar is used for sintering.

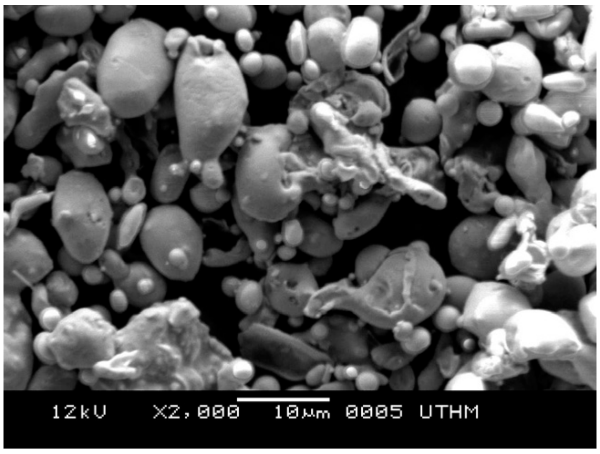

Figure 1. Morphology of the SS316L water-atomised powder.

Table 1. SS316L water atomised powder particle size distribution.

	D_{10}	D_{50}	D_{90}	S_w	S (m^2/g)
Coarse	4.985	15.052	34.747	3.036	0.573
Fine	3.338	7.157	17.515	3.588	0.978

3. DESIGN OF EXPERIMENT

There are many moulding parameters that have some effects on the properties of the green part quality. Therefore, a design of experiment (DOE) methods is necessary for the experimental work involving several inputs. The most frequently used methods are partial or full factorial design and the Taguchi approach. With an appropriate DOE, one can quickly and with fewer number of trials, find out whether the variables have an effect on the output quality. The Taguchi approach is mostly used in the industrial environment, but it can also be used for scientific research. The method is based on balanced orthogonal arrays [12]. In this paper, L_{27} (3^{13}) orthogonal array consisting of 27 experiment trials and 13 columns are used to obtain the signal to the noise ratio (S/N) of every green part quality. Consequently, the S/N ratio is analysed in ANOVA form to determine the significant level and contribution of each variable to the green part quality.

Considering the interaction between the main factors that influence the quality of the green part, three interactions have been identified from previous work [5-8]. The main variables involved in this study are as shown in Table 2. Three levels for each variable refer to the maximum and minimum limits that may influence the quality of the green part.

4. Results and Discussion

The analysis of variance (ANOVA) of the S/N ratio is shown in Table 3. Factors with low variance, v_n are pooled together as error, e and only those with a significant level less than $\alpha = 0.1$ are accounted in the ANOVA. The ANOVA table for minimising green defects is shown in Table 3 (a). The holding time is found to be highly significant at $\alpha = 0.025$ for minimising the green defects

Table 2. Factor level (variables) in the experiment.

Level	Moulding Pressure, A (bar)	Moulding Temperature, B (°C)	Mould Temperature C (°C)	Holding Pressure, D (bar)	Moulding rate, E (ccm/s)	Holding time, F (s)	Cooling time, G (s)
0	550	150	50	800	5	5	2
1	650	155	55	1000	10	10	6
2	750	160	60	1200	15	15	10

Table 3. ANOVA table for the S/N ratio.

a. Minimising green defects

Factor	Degree of freedom, f_n	Sum squared, S_n	Pure Sum squared,	Variance, v_n	Variance ratio, F_n	Contribution, P_n
A	2	6.516	pooled			
B	2	2.852	pooled			
C	2	28.044	21.66287	14.022	4.394836**	12.73
D	2	4.202	pooled			
E	2	20.811	14.42987	10.4055	3.261337±	8.48
F	2	38.468	32.08687	19.234	6.028403*	18.86
G	2	3.135	pooled			
A × B	4	31.792	19.02975	7.948	2.491096±	11.18
A × C	4	17.829	pooled			
B × C	4	16.515	pooled			
Error, e	16	51.049		3.190563		48.75
Total:	26	170.164				100

‡: $\alpha = 0.01$; *: $\alpha = 0.025$; **: $\alpha = 0.05$; ±: $\alpha = 0.1$

b. Maximising the green density

Factor	Degree of freedom, f_n	Sum squared, S_n	Pure Sum squared,	Variance, v_n	Variance ratio, F_n	Contribution, P_n
A	2	0.043958	0.0368368	0.021979	6.172836039‡	26.02
B	2	0.019293	0.0121718	0.009646	2.709093973±	8.60
C	2	0.014874	pooled			
D	2	0.010905	pooled			
E	2	0.001711	pooled			
F	2	0.011314	pooled			
G	2	0.002385	pooled			
A × B	4	0.011512	pooled			
A × C	4	0.011275	pooled			
B × C	4	0.014358	pooled			
Error, *e*	22	0.078334	0.003560636			65.39
Total:	26	0.141585				100

‡: $\alpha = 0.01$; *: $\alpha = 0.025$; **: $\alpha = 0.05$; ±: $\alpha = 0.1$

c. Maximising the green strength

Factor	Degree of freedom, f_n	Sum squared, S_n	Pure Sum squared,	Variance, v_n	Variance ratio, F_n	Contribution, P_n
A	2	0.2663	Pooled			
B	2	1.2845	Pooled			
C	2	9.1054	8.252355	4.5527	10.67399□	35.01
D	2	0.3963	Pooled			
E	2	1.723	Pooled			
F	2	5.0799	4.226855	2.53995	5.955017□	17.93
G	2	0.9184	Pooled			
A × B	4	1.3555	Pooled			
A × C	4	1.6689	Pooled			
B × C	4	1.7706	Pooled			
Error, *e*	22	9.3835	0.426523			47.05
Total:	26	23.5689				100

‡: $\alpha = 0.01$; *: $\alpha = 0.025$; **: $\alpha = 0.05$; ±: $\alpha = 0.1$

followed by the mould temperature at $\alpha = 0.05$ and the moulding rate which is at the lowest significant level ($\alpha = 0.1$). The interactions between moulding pressure and moulding temperature have shown a low significant level at $\alpha = 0.1$ with a contribution to minimise the green defects to only 11.18 % compared to the holding time at 18.86 %.

Moreover, the ANOVA table for maximising the green density, as shown in Table 3 (b), demonstrates that only moulding pressure and moulding temperature are significant to the green density. Moulding pressure demonstrates the utmost significant level (α = 0.01) while moulding temperature is only significant at α = 0.1. In addition, ANOVA for maximising the green strength as shown in Table 3 (c) shows both mould temperature and holding time are highly significant (α = 0.01) for the green strength. These variables contribute about 35.01 % and 17.93 % respectively to the green strength.

By considering only factors with high significance levels, the simultaneous optimal parameter for this moulding process is summarised in Table 4. As shown in Table 4, the optimum factor level that satisfies the green part quality is $A_2B_1C_0E_1F_2$ and the optimal moulding parameters are as follows: Moulding pressure: 750 bar; moulding temperature: 155°C; mould temperature: 50°C; moulding rate: 10 ccm/s; and holding time: 15 seconds. However, holding pressure and cooling time are not significant for the quality of the green part.

Figure 2 demonstrates that the sintered density of the specimens moulded using the optimal moulding parameters that are presented in this paper. The sintered density increases gradually when the sintering temperature increases. In this study, the heating rate remains at 10°C/minute. This is based on an earlier studies published by Omar [5] and Suri et al. [13] that such heating rate is sufficient for sintering a SS316L compact. Prior to reaching the sintering temperature;

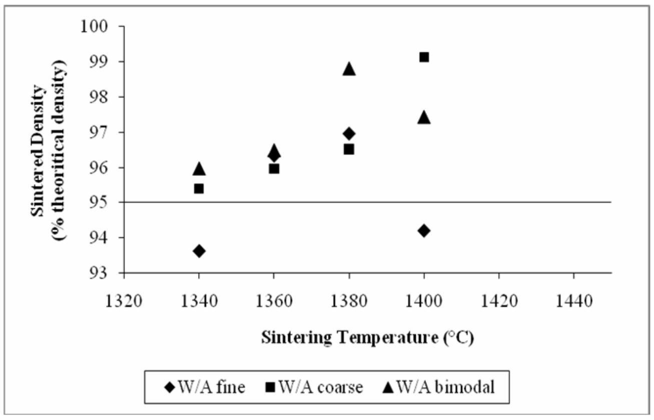

Figure 2. Compact sintered density. Heating rate and cooling rate: 10°C/min; thermal pyrolysis: 600°C for 20 min; dwell time: 4 hour.

Table 4. Significant level of the simultaneous optimisation.

Variables		Factor level	Significant level, α
A	Moulding pressure	A2	0.025
B	Moulding temperature	B1	0.1
C	Mould temperature	C0	0.01
D	Holding Pressure	Not significant	
E	Moulding rate	E1	0.1
F	Holding time	F2	0.01
G	Cooling time	Not significant	
A × B		A2 B2	0.1
A × C			
B × C			

pre-sintering is performed at 600°C in 20 minutes under the same atmosphere to decompose remaining binders left by the thermal pyrolysis [14].

The minimum limit shown in Figure 2 refers to the minimum MIM compact density suggested by German and Bose [3]. As shown in Figure 2, sintering temperature at 1340°C is unable to provide a better sintered density to the fine powder compact. This is due to the fact that the irregular shape of the water-atomised powder particles provides more pores to the compact besides limiting the production of the green part at a higher powder loading with this irregular shaped fine powder. As reported by Jamaludin et al. [15], the optimum powder loading of this powder is at a low volume of 62.5 %. This is by the fact that as shown in Figure 3, more pores appear on the Scanning Electron Microscopy (SEM) image of the fine powder compact sintered at 1,340°C.

Low critical powder loading on the fine water-atomised powder is also derive from the large surface area own by the fine powder particle, causing more inter-particle friction between fine powder particles [16]. This is shown in Table 1 that the specific surface area,

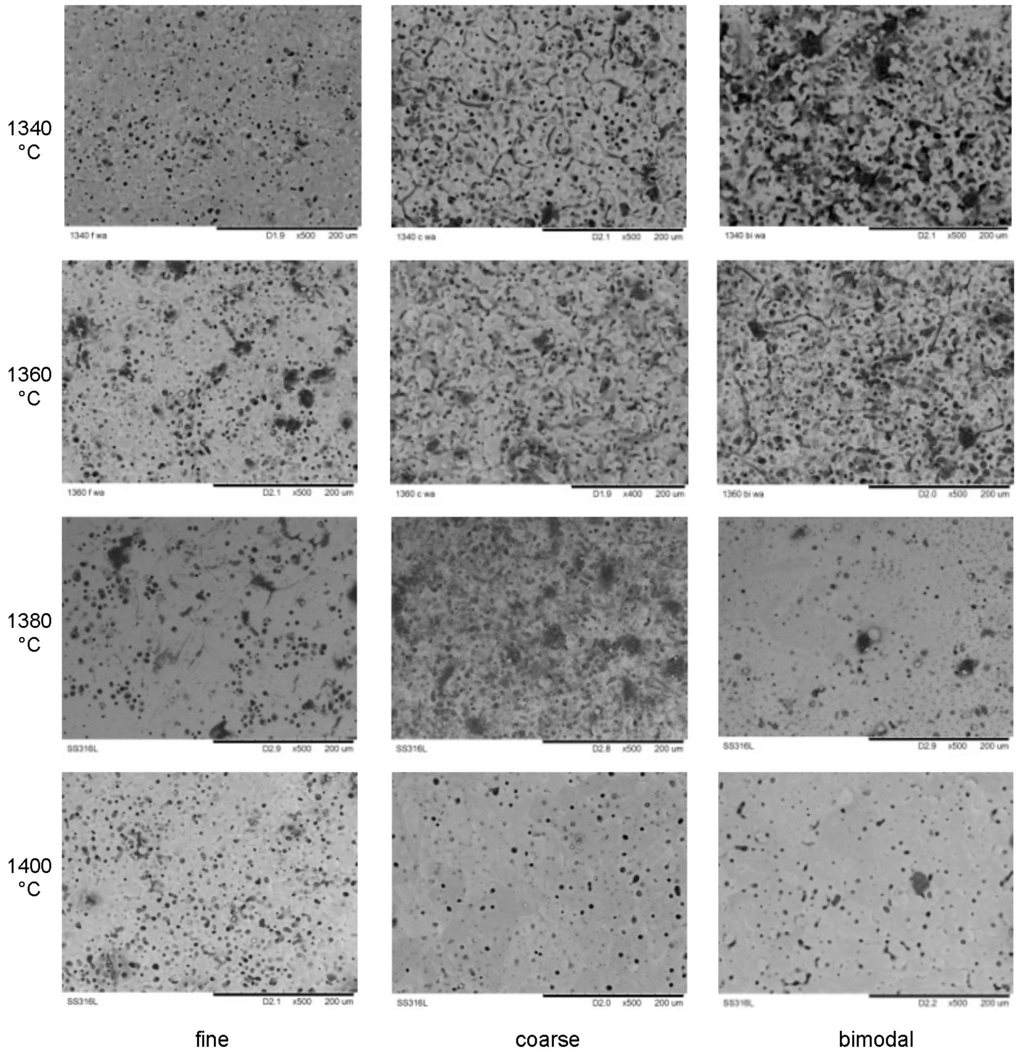

Figure 3. The Scanning Electron Microscopy (SEM) image of the sintered compact.

S of the fine powder particle is larger than the coarse powder, 0.978 m^2/g and 0.573 m^2/g respectively. Another reason that limits the optimal powder loading of this powder to 62.5 % also resided to the irregular and ligamental shape of the powder particles that shown in Figure 1. At the mean time, Figure 2 shows that bimodal water-atomised compact dominates the sintered density as compared to the monomodal compacts. Bimodal compacts attain the highest sintered density at sintering temperatures of 1,340°C, 1,360°C and 1,380°C. However, a reversed situation occurs at 1,400°C. Sintered density of a fine powder compact plummets from 96.96% to 94.21% of the theoretical density while sintered density of water-atomised bimodal compact also slumps from 98.81% to 97.44% of the theoretical density when sintering temperature rises to 1,400°C. Nevertheless, sintered density of the coarse powder compact remains high at 99.14% of the theoretical density. This is by the fact that the porosity of the compact is increasing due to the pore coarsening when the sintering temperature exceeded the melting temperature, as this happens, liquid start to appear in the powder matrix. German [17] breaks the persistent liquid phase sintering process into three stages: (1) initial stage: solubility and rearrangement, (2) intermediate stage: solution and reprecipita-tion, and (3) final stage: microstructural coarsening. These stages overlap and are characterised by different microstructural phenomena that clearly described by German [l7]. Among those phenomena, two are relevant: densification and microstructural coarsening. After liquid formation, densification occurs through primary and secondary rearrangement and solution and reprecipitation. The densification rate decreases with sintering time until a rigid skeleton is formed in the material and pore stabilization by entrapped atmosphere occurs. At this point densification ends. At the same time, the exposure of the material to high temperature tends to induce grain growth, driven by the excess of free energy associated with the grain boundary surface. Densification starts earlier than grain growth, the latter occurring in particular for prolonged sintering processes. In addition to the evolution of pore morphology and amount, an important microstructural phenomenon is grain coarsening [l7]. This is activated by the liquid; therefore, the persistence of liquid during the entire LPS process may worsen some mechanical properties as an example the density.

Reduction of sintered density occurs due to excessive liquid phase in the powder matrix during sintering at 1,400°C, especially when the powder loading is too low in the fine water-atomised powder compact. Liquid phase is expected to exist in SS316L when the sintering temperature reaches 1,350°C and 1,390°C [14]. Melting temperature of SS316L is 1,375°C [17]. However, liquid phase in solids will enhance the densification process, but too much liquid will reduce sintered density due to microstructural coarsening [18]. Particle melting occurs during liquid phase sintering, resulting in a solid-liquid mixture during the thermal cycle. The liquid phase provides bonding, contributes a capillary force, and usually enhances the rate of mass transport as compared to solid-state process. Furthermore, coarse powder compact sintered at 1,400°C improves its density to 99.14 % of theoretical density. This is due to the small surface area that eliminates liquid phase in the compact. A small surface area of powder particles delays the liquid phase formation although the sintering temperature is already exceeding the melting temperature of the materials. This occurs due to the fact that less inter-particle contact takes place as the small surface area delays surface energy reduction. In addition less pores is illustrated by the SEM in Figure 3 for the coarse powder compact, sintered at 1,400°C. However as the sintered density of the fine powder compact plummet at 1,400°C, thus the SEM image in Figure 3 shows more pores on this compact compared to the coarse powder compact.

5. Conclusions

The Taguchi and ANOVA methods are very helpful in determining the importance of variables when optimising a quality characteristic including, in this case, the green defects, green strength and green density of a MIM compact. Based on the investigation, the following conclusions can be made regarding the green part quality and the compact densification.

- Mould temperature and holding time are the most significant variables to attain best green part quality.
- Optimised moulding parameter enables to improve the green part quality and thus improves the sintered density.
- Bimodal gas-atomised particle size distribution compacts attain better densification than reported by Omar [5] and German [19].
- Bimodal water-atomised compacts dominate the sintered density compared to the monomodal compacts except at sintering temperature of 1,400°C.
- Sintered density of a fine and bimodal water-atomised powder compacts plummet when sintered at 1,400°C.

Acknowledgement

Our deepest appreciation and gratitude to National University of Malaysia for the research grant, UKM-KK-02-FRGS0013-2006.

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